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ORIGINAL REPORT



Ultrahigh GaN:SiO₂ etch selectivity by *in situ* surface modification of SiO₂ in a Cl₂-Ar plasma

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ABSTRACT

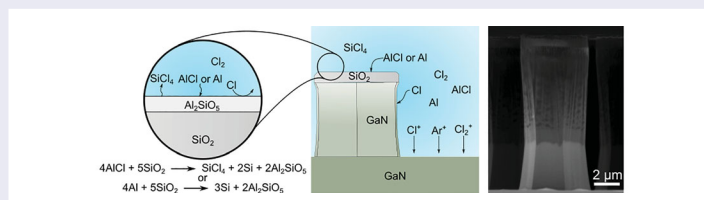
The realization of vertical GaN devices requires deep plasma etching and is contingent on high mask selectivity. In this work, we show that SiO₂ can be an effective mask material for deep etching GaN with GaN:SiO₂ selectivities greater than 40—higher than the conventionally reported 15 for metal hard masks such as nickel. Ultrahigh SiO₂ selectivities were achieved by introducing Al and AlCl into the Cl₂-Ar inductively coupled plasma, which reacts with the SiO₂ mask surface to form an etch-resistant aluminum silicate surface layer. This mechanism provides a low-contamination pathway to etch deep GaN microdevices.

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Plasma etching; gallium nitride; wide bandgap



IMPACT STATEMENT

This manuscript reports on exceptionally high GaN:SiO₂ etch selectivities in a Cl₂-Ar plasma which will enable fabrication of new classes of GaN device structures that currently only exist in theory.

Introduction

Gallium nitride (GaN) is a wide bandgap semiconductor used ubiquitously in the lighting and RF switching industries. It is considered a successor to silicon for many power applications due to its superior properties such as a wider bandgap for higher temperature operation [1], higher electron mobility for faster switching [2], greater critical electric field for higher voltages [3], and direct bandgap for optoelectronics [4]. Many realized [5–9] and proposed devices such as superjunctions [10–12] promise to revolutionize power conversion and transmission but require highly selective etch processes to realize their device geometries. There are no practical wet etchants for selective anisotropic etching of GaN [13], and like other wide bandgap semiconductors, it is difficult to plasma etch without creating rough and damaged surfaces [14]. Typical etch recipes rely on energetic ion bombardment to improve etch rates, which results in poor mask selectivities [15–17]. For deep etching, metal

hard masks such as nickel (Ni) are used to achieve selectivities on the order of 15 [16,18,19]. However, Ni can contaminate the fabricated chip and downstream semiconductor fabrication processes [20,21], can be challenging to pattern in thick films required for deep etching of fine features, and can result in roughness around the mask edges due to the finite grain size of metal films [22]. To avoid these issues, conventional dielectric layers like SiO₂ with mature deposition and patterning processes for thick films (e.g. 1 μm or thicker) could be employed. Unfortunately, GaN:SiO₂ selectivities reported in the literature are low at ~6 [15–17], which are not ideal for deep etching. Previously, we reported exceptionally high GaN:SiO₂ etch selectivities (39:1) using a Cl₂-Ar plasma in an Electron Cyclotron Resonance (ECR) plasma etcher [23]. The goal of this work is to determine the cause of this unusually high selectivity.

Previously, we detected Al-based etch residue on etched feature sidewalls of samples etched in the ECR

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system, which was endemic due to the use of an Al carrier plate. We hypothesized that the high etch selectivities can be attributed to Al reacting with the SiO₂ mask to form an aluminum silicate (Al₂SiO₅) surface layer on the SiO₂, which reduces the mask etch rate. Here, we expand on this data by modulating the amount of Al and AlCl introduced into a Cl₂-Ar inductively coupled plasma (ICP) by placing GaN chips on either a sapphire, aluminum nitride (AlN), or Al wafer during the etch. We examined the plasma species using optical emission spectroscopy (OES) and analyzed the surfaces of the etched sample by Auger electron spectroscopy (AES) to measure etch by-products. We confirm that GaN:SiO₂ etch selectivities over 40 can be achieved by *in situ* formation of an Al₂SiO₅ surface layer. These high selectivities open possibilities for the fabrication of a new class of unrealized devices, which require low-contamination deep etching of GaN.

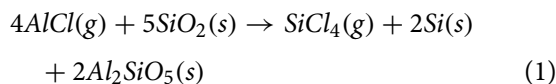
Experimental

20 μm thick GaN layers grown on (0001) sapphire were used in this etch study. A 1 μm thick SiO₂ mask layer was deposited using a Plasma-Therm plasma-enhanced chemical vapor deposition system at 320°C with 400 sccm 2% SiH₄/N₂, 900 sccm N₂O, 40 W RF power, and 900 mTorr. Prior to the SiO₂ deposition, the GaN was sequentially cleaned by sonication for 5 min in acetone, isopropyl alcohol, and methanol followed by self-heated piranha solution (3:1 H₂SO₄:H₂O₂) for 15 min. SiO₂ circles 2–10 μm in diameter were patterned using photolithography and dry etched in a C₄F₈-Ar plasma. The wafers were then diced to 7 × 7 mm² chips and cleaned again by sonication in solvents followed by piranha.

The chips were etched in an Oxford Instruments PlasmaPro 100 ICP etcher. The chips were placed on a carrier wafer (sapphire, AlN or Al) without thermal paste and loaded into the etch chamber. The plasma etch conditions were 28 sccm Cl₂, 3.5 sccm Ar, 2 mTorr, 1000 W ICP power, with the table temperature at 200°C, and 25 W or 75 W RF power. The GaN etch rate was measured by scanning electron microscopy (SEM). The SiO₂ etch rate was measured using spectroscopic reflectivity measurements using a NanoSpec TOHO 3100 on SiO₂-coated silicon chips which were loaded alongside the GaN chips and exposed to the same etch plasma. Plasma compositions were characterized by optical emission spectroscopy (OES) using an Ocean Optics Flame spectrometer. A PHI 710 scanning Auger nanoprobe was used to analyze the surface composition of the etched samples.

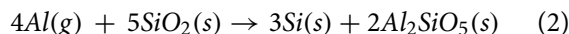
Results and discussion

Previous experiments on deep etching GaN in an electron cyclotron resonance (ECR) plasma etcher resulted in anomalously high GaN:SiO₂ etch selectivities of 39 [23]. After etching in that system, the etched features were visibly coated with a residue as seen in the SEM image in Figure 1(b) which were revealed to be an Al-containing material by Auger electron spectroscopy (Figure 1(a)). The Al residue was easily removable by solvent or 3:1 H₂SO₄:H₂O₂, and a cleaned pillar is depicted in the inset of Figure 1(b). We determined that the likely source was the Al carrier plate. We also hypothesized that the Al residue was responsible for the increased selectivity by the mechanism depicted in Figure 1(c). Volatile AlCl or sputtered aluminum released from the plate is transported through the plasma to the GaN sample surfaces. When AlCl comes into contact with the SiO₂ mask, the following reaction takes place to transform the SiO₂ into Al₂SiO₅ [24]:



$$\Delta G_{rxn}^o = -400 \frac{\text{kJ}}{\text{mol}} \text{ of } \text{Al}_2\text{SiO}_5$$

If elemental Al contacts the SiO₂ surface, then the following reaction can proceed:



$$\Delta G_{rxn}^o = -293 \frac{\text{kJ}}{\text{mol}} \text{ of } \text{Al}_2\text{SiO}_5$$

Based on the values in the NIST-JANAF Thermochemical Tables, both reactions are strongly thermodynamically favorable. The Si in the product of these reactions can be easily etched by free Cl species in the plasma while pure Al₂O₃ is known to etch more slowly in a Cl₂-Ar plasma [25,26]. For SiO₂, etching is rate limited by the removal of O by physical sputtering [27]. The threshold displacement energy for O in Al₂O₃ (~75–80 eV) [28–30] is much stronger than that of SiO₂ (~10–50 eV) [31,32] and therefore sputtering is expected to be lower for Al₂O₃ compared to SiO₂. Although plasma etching of Al₂SiO₅ compounds are unexplored in the literature, since Al₂SiO₅ and other aluminum silicates are composed of Al₂O₃ and SiO₂ polyhedra (the base building blocks of each compound), the etch rate may be expected to be between that of pure Al₂O₃ and SiO₂ and be rate limited by etching of the Al₂O₃ polyhedra. Additionally, adding small amounts of O₂ to Cl₂-based plasmas results in high GaN:AlGaN selectivities as high as 68:1 through

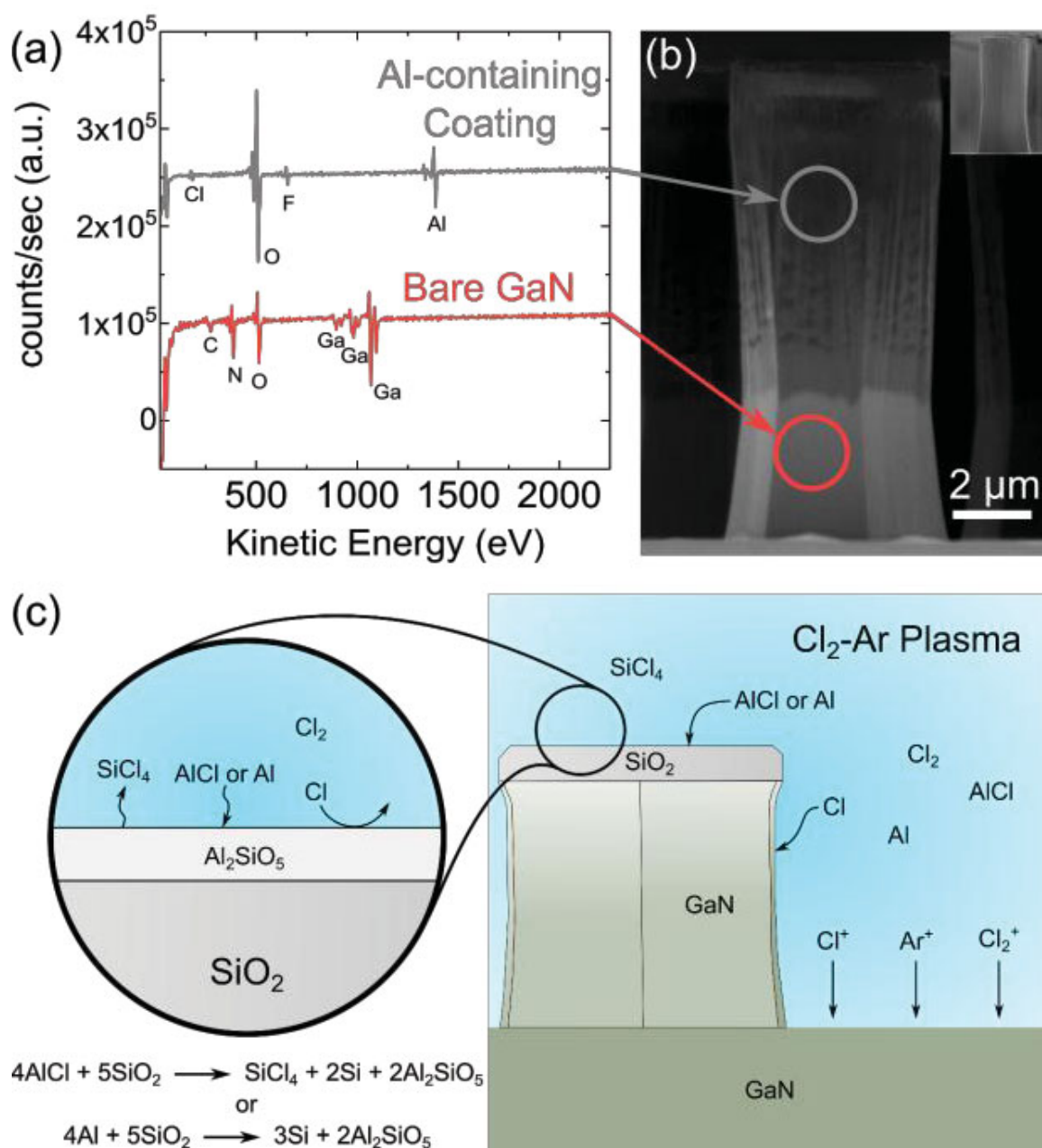


Figure 1. Auger electron spectra (a) of a GaN pillar (b) etched in an electron cyclotron resonance (ECR) plasma etcher showing the presence of an Al-containing residue on the pillar after etching. Schematic of the mask hardening mechanism (c). Al-containing species in the plasma react with the SiO_2 surface to form a robust, etch-resistant Al_2SiO_5 surface layer which enhances the effective GaN: SiO_2 etch selectivity.

the formation of Al_xO_y compounds on the AlGaN surface [33–36]. Thus, the reaction of Al or AlCl with the SiO_2 surface is expected reduce the mask etch rate, and therefore increases the GaN: SiO_2 selectivity. We believe that this is the cause of our anomalously high selectivity in our prior results.

To test our hypothesis, we modulated the amount of Al entering a Cl_2 -Ar plasma in an inductively coupled plasma etcher while etching GaN. The introduction of Al to the plasma was done by placing the GaN chips onto

carrier wafers of different Al-containing materials which etch incidentally in the plasma. OES of the plasma verifies the relative amounts of Al in the plasma (Figure 2) when using the different carrier wafers. The sapphire releases a negligible amount of Al in the plasma as is indicated by only a small Al peak at 396 nm [37] (note: OES of a fused silica wafer (not shown) produces a nearly identical spectrum with a small Al peak at 396 nm). Thus, the sapphire OES spectrum can be effectively taken as the background spectra of the Cl_2 -Ar plasma). The AlN wafer produces a

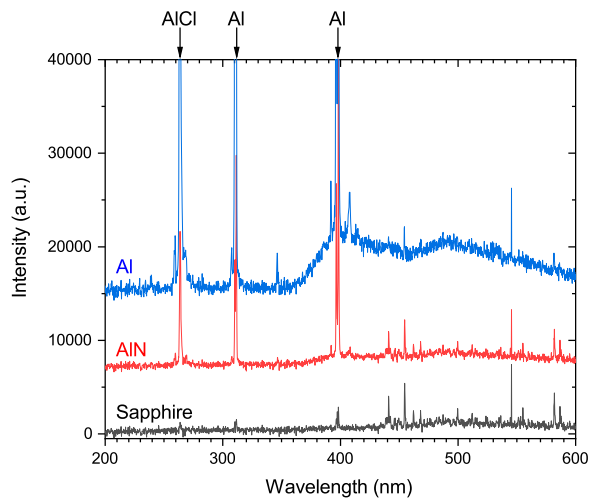


Figure 2. Optical emission spectra of Cl_2 -Ar plasmas (25 W RF power) using sapphire, AlN, and Al carrier wafers. Based on the spectra, sapphire releases a negligible amount of free Al or AlCl into the plasma. While the AlN and Al release moderate and high amounts of Al and AlCl into the plasma, respectively.

strong AlCl signal at 261 nm [38–40] and strong Al signals at 308 and 396 nm [37] in the OES. The Al wafer has even stronger AlCl and Al peaks in the OES than the AlN indicating that it releases more Al species into the plasma. Thus, the relative amounts of Al containing-species in the plasma resulting from the carrier wafers is sapphire < AlN < Al.

Figure 3 shows the GaN and SiO_2 etch rates, and the GaN: SiO_2 etch selectivity for samples etched using each of the carrier wafers. Figure 4 depicts cross-sectional SEM images of the etched GaN using the various carrier wafers, which highlights the significant impact of the carrier wafer and RF power conditions on the etch rates and surface morphology. The etch rates for both the GaN and the SiO_2 are higher for the 75 W RF power condition (DC bias ≈ -205 V), as expected due to higher ion bombardment energy. At 25 W RF (DC bias ≈ -87 V), the etch rate is reduced by about a factor of six or more when

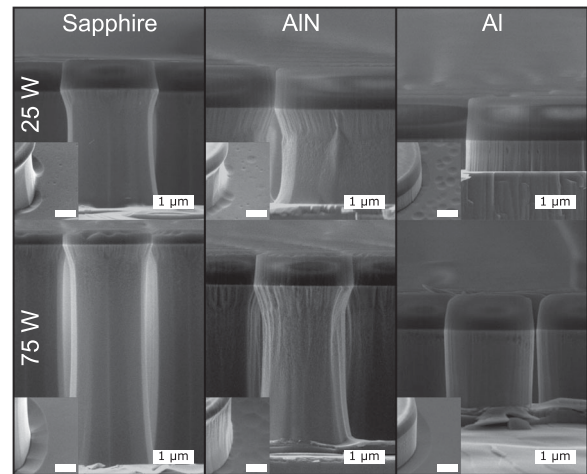


Figure 4. Scanning electron images of the etched GaN pillars using sapphire, AlN, and Al carrier wafers at 25 and 75 W RF power. The insets show the etched floor imaged at a 45° angle. The scale bar in the inset is 1 μm .

using AlN and Al compared to sapphire. The lower GaN etch rate for AlN and Al may be due to sinking of the Cl_2 in the plasma. Despite the lower GaN etch rates for these materials, the extremely low SiO_2 etch rate results in much higher etch selectivities. These results support our hypothesis that Al species in the plasma are increasing the GaN: SiO_2 etch rate by slowing the SiO_2 etching. The carriers also have an effect on the surface morphology of the etched features. Trenching is influenced by the carriers and is present to some extent under each condition and can be seen in the insets in Figure 4. This phenomenon is a result of ions glancing off of the etched side walls and increasing the ion flux near the base of the etched features [41]. Pitting is also observed on the etched floor, particularly under 25 W RF power. These etch pits are a result of defect selective etching and are more pronounced under a more chemically driven etch (lower ion energy) at 25 W RF.

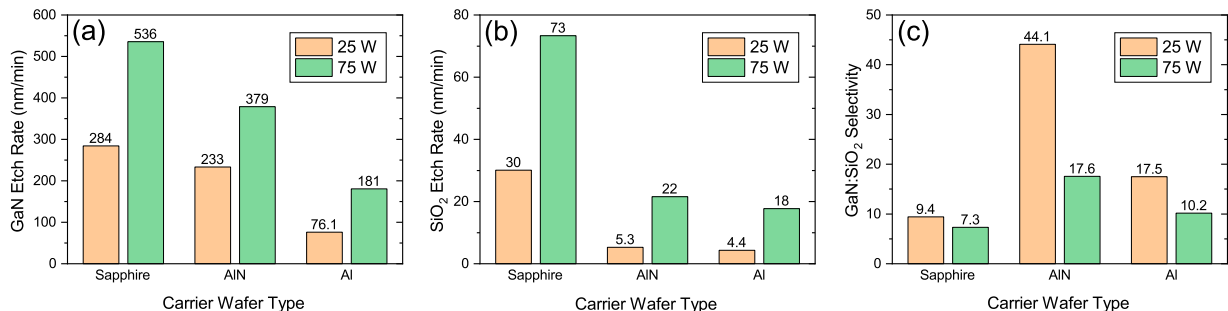


Figure 3. GaN etch rates (a) SiO_2 mask etch rates (b) and subsequent GaN: SiO_2 etch selectivity (c) for the samples etched using different carrier wafer types at 25 and 75 W RF power. When AlN and Al carrier wafers are used, the SiO_2 etch rate is drastically reduced, resulting in higher GaN: SiO_2 etch selectivities compared to using the sapphire carrier wafer.

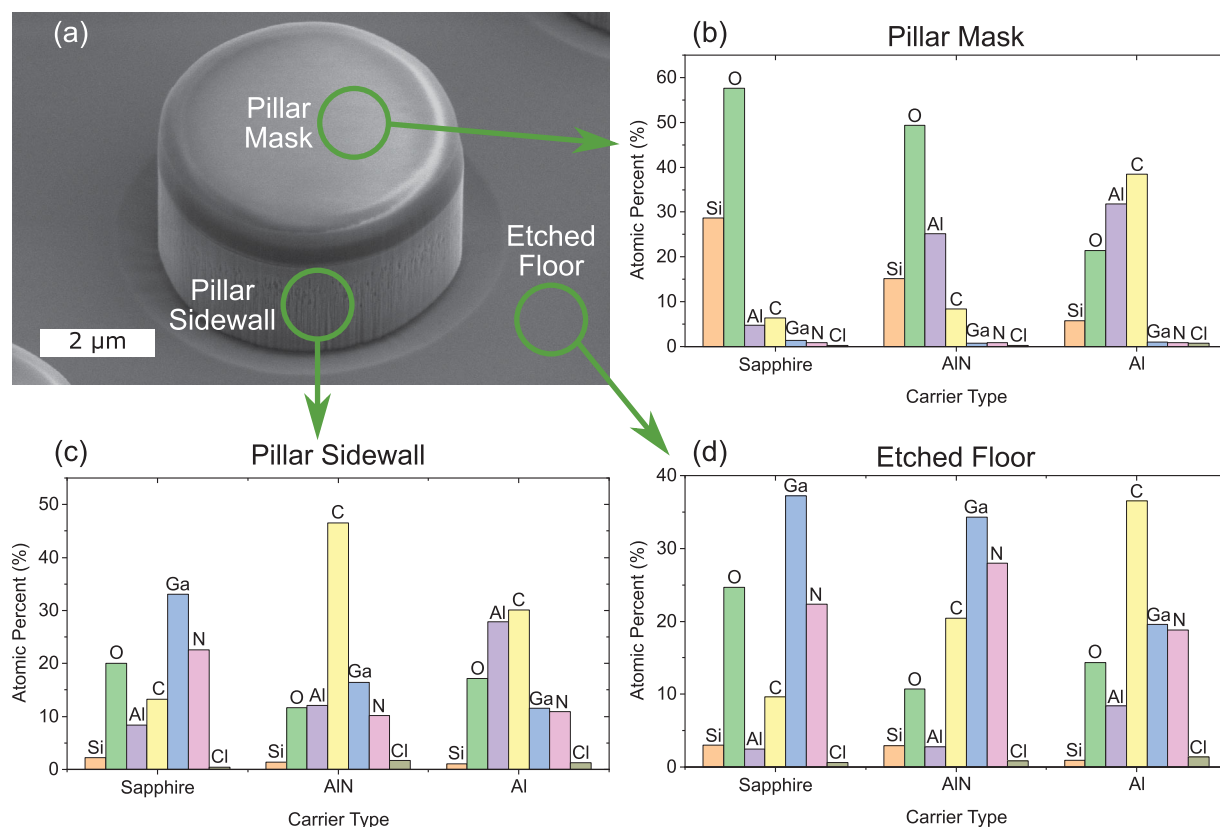


Figure 5. Scanning electron image of GaN pillar etched at 25 W RF power (a) and atomic surface composition of the pillar mask (b), pillar sidewall (c), and the etched floor (d) as measured by Auger electron spectroscopy. The surface composition of the mask reveals that Al released from the AlN carrier wafer reacts with the SiO_2 to form an Al_2SiO_5 surface layer while the SiO_2 surface for the sapphire carrier wafer remains as SiO_2 .

To confirm the presence of an Al_2SiO_5 layer on the surface of the SiO_2 masks due to Al species in the etch plasma, AES was measured on the pillar tops (on the SiO_2 mask), the pillar sidewall, and the etched floor. The compositions of the surfaces are shown in Figure 5. The mask of the sapphire sample is composed largely of Si and O, indicating that the surface is unmodified. In contrast, the mask surface of the AlN sample has a reduced Si concentration and is enriched in Al. The measured composition is $\text{Al}_{0.28}\text{Si}_{0.17}\text{O}_{0.55}$, which is in reasonable agreement with the formula Al_2SiO_5 . The lack of a strong Al signal on the etched floor indicates that the Al is indeed reacting with the SiO_2 and is not easily sputtered from the surface. The Al carrier wafer coated all surfaces with an Al and C-rich material. The presence of Al on the mask likely results in a similar surface reaction with the SiO_2 , but such a layer would be obfuscated by the large amount of Al and C on the surface. The excessive coating of all of the surfaces explains the low GaN etch rate (the coating on the etched floor needs to be continuously removed to etch the GaN) and the vertical sidewall (the coating passivates the surface and prevents lateral etching of the GaN).

The surface composition of the SiO_2 mask correlates with both the OES signal of Al in the plasma (Figure 2) and the etch rate of the SiO_2 (Figure 3). When the sapphire carrier wafer is used, insignificant amounts of Al enter the plasma, and the SiO_2 mask etch rate is unaffected. If either the AlN or Al carrier wafer is used, AlCl and Al are introduced into the plasma which can then react with the SiO_2 to form a hardened Al_2SiO_5 surface layer which slows the mask etch rate and increases the GaN: SiO_2 etch selectivity. Despite the etch resistance of the surface modified mask to the Cl_2 -Ar plasma, the masks are still easily removed by wet etching using buffered oxide etch (BOE) for 15 min, consistent with reports of atomic layer deposited Al_2O_3 [42] and Al_2SiO_5 [43].

For chip-level processing, using an etchable Al-containing carrier to introduce Al directly to the plasma is an easy and accessible way to increase GaN: SiO_2 mask selectivity. However, as the chip size is increased and more of the carrier is covered, uniformity may become a concern as the Al needs to be transported from the carrier, into the plasma, and back to the GaN surface. Further, as less of the carrier is exposed to the plasma,

less Al will be introduced to the plasma, and the process would be affected. Ultimately, to overcome these concerns, a more controllable method for introducing Al into the plasma should be pursued. This may be as simple as bleeding small amounts of an Al-containing gas such as AlCl, AlCl₃ or trimethylaluminum (TMA) continuously into the plasma. These Al sources would then be treated as a process gas and the amount of Al in the plasma could be directly and independently controlled. Such a technique would provide a scalable method to take advantage of standard SiO₂ deposition and patterning techniques with the benefit of having an etch-resistant aluminum-silicate hard mask.

Conclusions

We understand and have demonstrated drastically improved GaN:SiO₂ etch selectivities in a Cl₂-Ar plasma by introducing Al species into the plasma. The Al species react with the SiO₂ surface to form an Al₂SiO₅ which is more etch resistant than pure SiO₂. This effect provides a route to using SiO₂ as a mask for deep etching of GaN without the contamination or patterning concerns that using metal masks entail.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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